UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

CENTER FOR ENVIRONMENTAL MEASUREMENT AND MODELING
RESEARCH TRIANGLE PARK, NC 27711

OFFICE OF RESEARCH AND DEVELOPMENT

June 17, 2021

Ken Kloo, Director
NJ Department of Environmental Protection
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Subject: NJ DEP Report #10: Targeted Analysis of PFAS in Wastewater and Groundwater Samples Collected at the Solvay Specialty Polymers Plant in West Deptford, N.J.

Dear Mr. Kloo:

I am pleased to provide you with the attached laboratory report that includes targeted analysis (TA) results for per- and polyfluoroalkyl substance (PFAS) in water samples collected from wastewater and groundwater influent associated with the Solvay Specialty Polymers plant in West Deptford, NJ. This is the tenth in a series of reports prepared as a part of EPA Office of Research and Development's (ORD) collaboration with the New Jersey Department of Environmental Protection (NJ DEP) and EPA Region 2 on the study, "Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Substances (PFAS) in Environmental Media from an Industrialized Area of New Jersey." The enclosed Report #10 provides results of the occurrence of legacy PFAS based on analysis of 13 water samples collected on March 10 and April 6, 2021.

It is our understanding that this information was requested by NJ DEP to help in their ongoing investigation into the presence of PFAS in the environment near manufacturing facilities of interest. This request relates to our research capabilities and interests applying targeted and non-targeted analysis methods for discovery of the nature and extent of PFAS environmental occurrence that may be potentially associated with industrial releases. EPA continues to develop analytical methods for many PFAS compounds in various media including some of those included in this report. We are providing the results of our analysis as they become available.

We do not interpret exposure or risk from concentrations of PFAS in this report. The EPA does not currently have final health-based standards, toxicity factors, or associated risk levels for PFAS, other than perfluorooctanoic acid (PFOA), perfluorooctane sulfonate (PFOS), and perfluorobutanesulfonic acid (PFBS). While the data provided in the attached reports indicate the

presence (or lack) of PFAS in the Solvay wastewater samples, we do not have sufficient information to offer interpretations related to human or environmental exposure and risk.

Thank you for inviting us to be part of this effort that helps to further both EPA's and New Jersey's understanding of an important issue in the state. This is just one of many Agency efforts that demonstrates EPA's commitment to cooperative federalism.

If you have any questions or concerns, do not hesitate to contact me at (919) 541-5114 or via email at Watkins.tim@epa.gov or Brian Schumacher at (706) 355-8001 or via email at Schumacher.Brian@epa.gov. I look forward to our continued work together.

Sincerely,

Timothy H Watkins

Timothy H. Watkins
Director

Enclosure

CC:

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Detection, Evaluation, and Assignment of PFAS in Environmental Media from an Industrialized Area of New Jersey

Laboratory Data Report #10: Targeted Analysis of PFAS in Wastewater and Groundwater Samples Collected at the Solvay Specialty Polymers Plant in West Deptford, N.J.

Background. This report stems from a collaborative study with EPA ORD, Region 2, and NJ DEP entitled "Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Substances (PFAS) in Environmental Media from an Industrialized Area of New Jersey". NJ DEP assumed responsibility for the collection of samples and their shipment to the ORD laboratory. ORD was responsible for sample extraction and analysis of PFAS. ORD's analysis and support team for this data report are listed in Table 1.

Table 1. EPA Office of Research and Development analysis and report team.

Responsibility	Personnel
ORD Principal Investigators	Mark Strynar, James McCord
Laboratory chemistry	James McCord, Mark Strynar
Quality Assurance Review	Sania Tong-Argao
Management coordination and review	Brian Schumacher, Myriam Medina-Vera
Report preparation	Kate Sullivan

This 10th report includes results of targeted analysis for PFAS in 13 water samples collected by NJ DEP from wastewater and groundwater influent locations at the Solvay Specialty Polymers (Solvay) plant and from the Gloucester County Utility Authority (GCUA) located in West Deptford. GCUA receives Solvay wastewater. Samples were collected on March 10, 2021 with repeated sampling on April 6, 2021. Samples collected March 10 were delivered to the ORD laboratory in Research Triangle Park, NC on March 11, 2021 and the second set of samples was delivered April 7, 2021. The results also include results for trip spike blanks, field blanks, and duplicates collected during the two sampling events. The results provided in this report were analyzed by Dr. James McCord and Dr. Mark Strynar at ORD's laboratory in Research Triangle Park, NC.

Seven perfluorinated carboxylic acids (PFCAs) and three perfluorinated sulfonic acids (PFSAs) were quantitated using a targeted analysis approach. Characteristics of these compounds including the compound name, CAS registry number (CASRN), chemical formula, and monoisotopic mass are provided in Table 2. These same 10 PFAS are registered in EPA's CompTox Chemicals Dashboard[1] where additional information about these chemicals can be found. The CompTox identification number (DTXSID) is also provided in Table 2.

[1] U.S. EPA CompTox Chemicals Dashboard https://comptox.epa.gov/dashboard

Table 2. PFCA and PFSA Analyzed with Targeted Analysis in Samples Collected at the Solvay Plant in West Deptford, NJ. Samples analyzed by UPLC-MS.

Acronym	Chemical Name	Formula	CAS Registry Number	Monoisotopic Mass (g/mol)	DTXSID
PFBA	Perfluorobutanoic Acid	$C_4HF_7O_2$	375-22-4	213.9865	DTXSID4059916
PFPeA	Perfluoropentanoic Acid	$C_5HF_9O_2$	2706-90-3	263.9833	DTXSID6062599
PFHxA	Perfluorohexanoic Acid	$C_6HF_{11}O_2$	307-24-4	313.9801	DTXSID3031862
PFHpA	Perfluoroheptanoic Acid	$C_7HF_{13}O_2$	375-85-9	363.9769	DTXSID1037303
PFOA	Perfluorooctanoic Acid	$C_8HF_{15}O_2$	335-67-1	413.9737	DTXSID8031865
PFNA	Perfluorononanoic Acid	$C_9HF_{17}O_2$	375-95-1	463.9705	DTXSID8031863
PFDA	Perfluorodecanoic Acid	$C_{10}HF_{19}O_2$	335-76-2	513.9673	DTXSID3031860
PFBS	Perfluorobutane Sulfonate	$C_4HF_9SO_3$	375-73-5	299.9503	DTXSID5030030
PFHxS	Perfluorohexane Sulfonate	$C_6HF_{13}SO_3$	355-46-4	399.9439	DTXSID7040150
PFOS	Perfluorooctane Sulfonate	$C_8HF_{17}SO_3$	1763-23-1	499.9375	DTXSID3031864

METHODS IN BRIEF

Water samples were extracted and analyzed by ultra-performance liquid chromatography mass spectrometry (UPLC-MS) according to methods documented within an approved Quality Assurance Project Plan (QAPP)[2,3,4]. Methods are also generally described in McCord *et al,* 2019[5].

In brief, water samples (500 mL) were filtered and then extracted using a WAX solid phase extraction cartridge. PFAS was removed from the cartridge in methanol and the volume reduced to 1 mL under a gentle stream of dry nitrogen. An aliquot of the 1 mL concentrated sample was injected into a Thermo Fisher Scientific™ Vanquish™ UPLC coupled to a Thermo Fisher Scientific™ Orbitrap Fusion™ MS.

PFAS concentrations were determined using a targeted analysis approach based on authentic standard reference materials obtained from Wellington Laboratories Inc., Guelph, Ontario, Canada. Quantitation was based on mass-labeled internal multipoint calibration curves with a calibration range of 5 to 250 ng/L. These analyses were performed on unknown samples, laboratory blanks, and check standards. Samples with high concentrations were diluted as needed by factors ranging from 5 to 100x, with some samples processed at multiple dilutions. Samples

[2] U.S. EPA National Exposure Research Laboratory, Quality Assurance Project Plan: Detection, Evaluation and Assignment of Multiple Poly and Per-fluoroalkyl Substances (PFAS) in environmental media from an industrialized area of New Jersey. Prepared for New Jersey Department of Environmental Protection (NJDEP), L-PHESD-0031345-QP-1-0 (Previous ID: D-EMMD-IEIB-010-QAPP-01), September 14, 2017.

[3] U.S. EPA National Exposure Research Laboratory, Quality Assurance Project Plan: Targeted Analyses of Per- and Polyfluoroalkyl Substances (PFAS) in Liquids Samples. D-EMMD-0031917-QP-1-0, 06May2019.

[4] U.S. EPA Center for Environmental Measurement and Modeling, Amendment #3 to Quality Assurance Project Plan: Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Substances (PFAS) in Environmental Media from an Industrialized Area of New Jersey (L-PHESD-0031345-QP-1-3, 08Mar2021.)

[5] McCord, J., Strynar, M. Identifying Per- and Polyfluorinated Chemical Species with a Combined Targeted and Non-Targeted-Screening High-Resolution Mass Spectrometry Workflow. *J. Vis. Exp.* (146), e59142, doi:10.3791/59142 (2019). https://www.jove.com/video/59142/identifying-per-polyfluorinated-chemical-species-with-combined

from the two sampling dates were processed in two batches shortly after they were received at the RTP laboratory.

SUMMARY OF RESULTS

The samples were collected from the Solvay Plant wastewater effluent that is discharged either directly to surface water from their permitted NJPDES outfall (WWDSN001) or to the Gloucester County Utility Authority (WWDSN003), which also discharges to surface water (EFFGCUA). To evaluate the potential for PFAS to be present in Solvay's process source water, groundwater influent prior to treatment or fluorochemical processes was sampled (INFGW). Due to sanitary/industrial wastewater commingling concerns, a combined industrial effluent and sanitary sewer on the Solvay site that delivers wastewater to GCUA was also sampled (GCUA-PS). Due to these concerns raised by NJ DEP during its sampling on March 10, Solvay installed a sampling port on the pump that directly discharges industrial wastewater from the polymer process wastewater treatment system (V770-TREAT). According to Solvay, water at this sampling port eliminates any sanitary discharge interference and best represents the treated effluent from the polymer process as it exists prior to discharge to the GCUA. A sample was collected from this port during the April 6 sampling.

Sample concentrations expressed in ng/L for the 13 water samples identified by sample IDs assigned by NJ DEP are provided in Table 3. Table 3 combines results for samples collected on March 10 and April 6, 2021 with results organized by location. If sample dilution was necessary, the reported value was the lowest dilution needed to maintain concentration within the calibration range. If no peak areas were observed the concentrations are labeled ND. Sample concentrations are given as <LOD if there was an integratable peak but concentrations were too low to quantify. Samples less than 5 ng/L are less than the calibrated limit of quantitation (LOQ) and are flagged as "U". No samples exceeded the calibration range after appropriate dilution.

All 10 PFAS that were analyzed were detected at one or more sampling locations within the Solvay facility or at GCUA, but concentrations varied widely among PFAS. Concentrations of PFNA and PFOA were significantly greater than the other PFAS at several locations. Concentrations of PFDA, PFHxA and PFHpA were intermediate while the perfluorinated sulfonic acids (PFBS, PFHxS, and PFOS) were present in low concentrations or not detected. Concentrations of each analyte were similar between the two sampling events at each location.

All 10 PFAS analytes were present in the groundwater inflow (INFGW) samples and, with few exceptions, concentrations were similar to or significantly greater than the concentrations at the post process wastewater discharge locations. Relative concentrations between locations were generally similar for each analyte. For example, Figure 1 shows the average of the two samplings for PFNA, PFOA, and PFOS at each location.

SUMMARY OF QUALITY CONTROL MEASURES

Data were checked for compliance with a number of laboratory and field related quality control evaluation criteria as specified in the project QAPPs[2,3]. Quality control sample results are provided in Tables 3 or 4.

Table 3. PFAS Concentrations (ng/L) in Samples Collected at the Solvay Plant in West Deptford, NJ Determined by Targeted Analysis.

		PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFBS	PFHxS	PFOS
NJDEP Sample ID	**Sample Date**	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L
INFGW-1	3/10/2021	16.9	27.2	53.4	135	1,770 D10	12,600 D100	183	2.37 U	3.23 U	9.95
WWDUP-1[&]	3/10/2021	17.9	28.6	57.3	146	1,700 D10	11,700 D100	203	2.73 U	3.59 U	12.6
INFGW-2	4/6/2021	ND	19.9 D10	55.6	134	1,980 D10	15,200 D100	172	1.16 U	1.23 U	ND
WWDSN001-1	3/10/2021	10.3	14.3	27.1	64.8	751 D50	5,340 D50	86.2	1.82 U	2.88 U	9.74
WWDSN001-2	4/6/2021	ND	12.9	25.5	57.8	944 D10	6,300 D100	66.0 B2	0.11 U	1.12 U	ND
V770-TREAT	4/6/2021	1.44 U	<LOD	0.10 U	0.27 U	<LOD	2.29 U	1.76 U	<LOD	<LOD	0.81 U
GCUA-PS-1	3/10/2021	7.95	ND	2.56 U	1.85 U	11.6	503 D5	14.8	1.06 U	0.84 U	2.25 U
GCUA-PS-2	4/6/2021	ND	<LOD	<LOD	<LOD	1.45 U	101	2.00 U	<LOD	<LOD	0.08 U
WWDSN003-1	3/10/2021	ND	ND	ND	14.2	283 D10	1,570 D10	22.8	1.28 U	ND	3.20 U
WWDSN003-2	4/6/2021	ND	ND	3.27 U	9.51	229	1,780 D10	9.18 B2	ND	1.70 U	9.37
EFFGCUA-1	3/10/2021	12.2	13.4	28.7	7.16	22.1	30.0	4.06 U	26.8	3.63 U	8.98
EFFGCUA-2 (Rep 1)	4/6/2021	8.13	5.01	21.5	3.72 U	17.8	29.1	0.79 U	3.92 U	2.31 U	2.76 U
EFFGCUA-2 (Rep 2)	4/6/2021	6.77	4.82 U	21.6	3.14 U	17.8	30.7	0.45 U	3.95 U	2.28 U	2.82 U

QA/QC Flags

ND: Non-detect based on no integratable peak area

<LOD: Integratable peak but less than the limit of detection

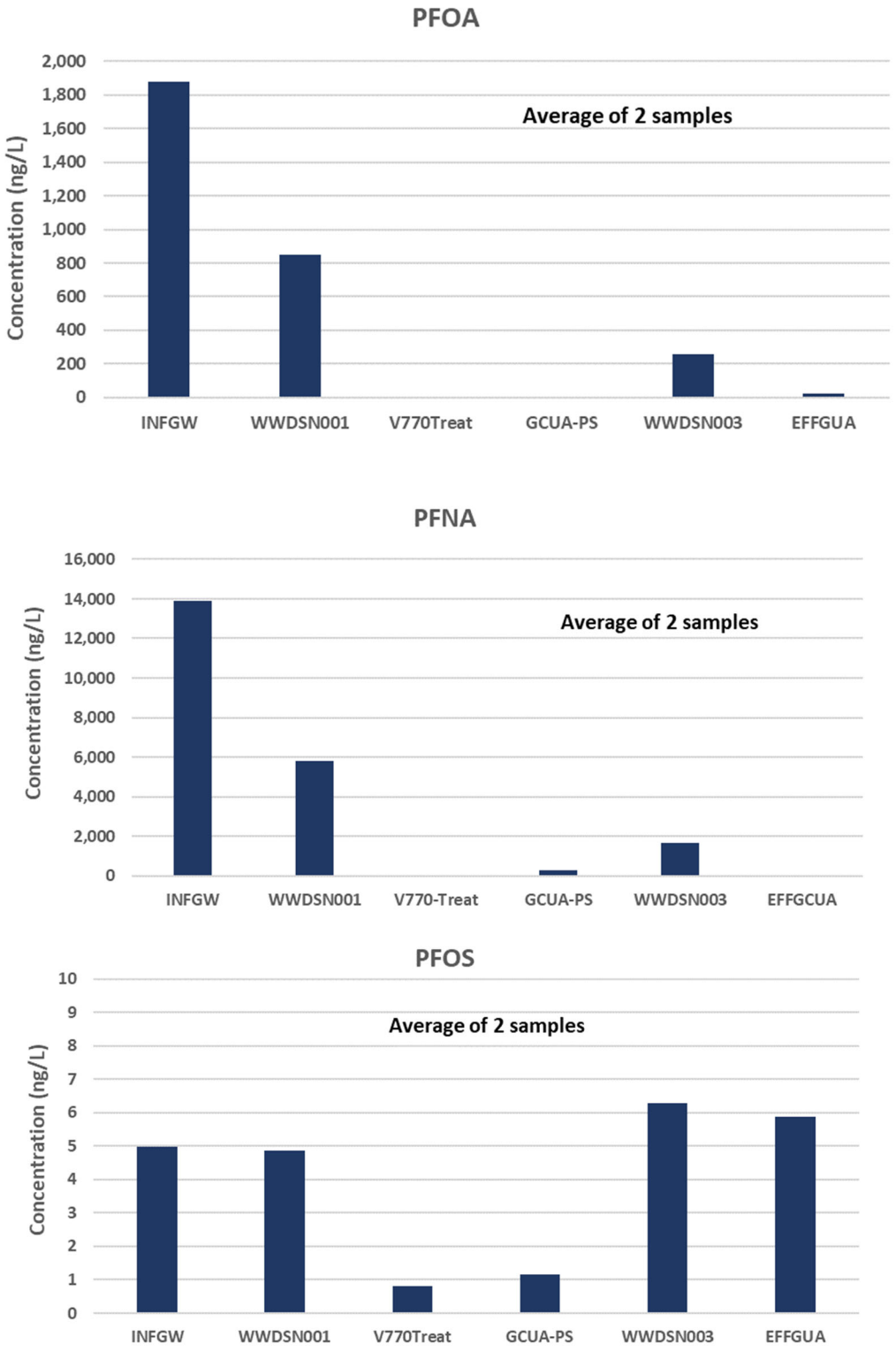
U: Less than the limit of quantitation; peak area detected but concentration less than the concentration of the lowest calibration standard (<5 ng/L)

Dx: Sample diluted by a factor (D5=5x, D10=10x, D50=50x, D100=100x)

B2 Analyte found in associated trip or field blank > LOQ and sample concentration is within 10x level of blank contamination; reported sample concentration may be elevated.

[&]Note that sample WWDUP-1 is a field duplicate paired with sample INFGW-1

Figure 1. Average concentration of two sampling events at various sampling locations.



CALIBRATION: 6-point calibration curves ranging from 5 to 250 ng/L were developed for each of the two batches of samples. The project QAPP specifies that back prediction of calibration curve points should be within ±30% for the lowest calibration point and ±20% of calibration for the rest of the calibration points, with the least-squares calibration line maintaining central tendency[3]. All analyte calibrations met criteria in both sample batches.

LABORATORY BLANKS. Analytes were not detected or were less than LOQ in both batches of laboratory blanks indicating no contamination (Table 4).

FIELD and TRIP BLANKS. Analyte concentrations in field blanks and trip blanks were generally either not detected or less than LOQ (Table 4). No PFAS were detected in the trip or field blanks carried during the March 10 sampling (Batch 1). However, PFDA was observed in concentrations exceeding LOQ in both the trip and field blanks in the April 6 sampling (Batch 2). Associated PFDA samples that are greater than LOQ but less than 10 times the concentration in the blanks are flagged as B2 in Table 3. Reported values for PFDA for April 6 samples in Table 3 are estimates and may be elevated due to contamination.

SPIKE BLANKS. One bottle spiked with 50 ng/L and 1 bottle spiked with 100 ng/L of the analytes were prepared by ORD, sent to NJ DEP in the week prior to sampling, and were carried to the field in each sampling event to evaluate analytical recovery. The ORD laboratory processed the spiked blanks in replicates of 2 or 3. The average concentration for each of the trip spike blanks is provided in Table 4. Spiked samples were generally within project goals of ±20%. The average deviation of blank concentrations from the spiked amount was -6.6% for the March 10 samples and 12.3% for the April 6 samples. The 50 ng/L trip spike collected April 6, 2021 failed to meet recovery criteria for PFDA and is flagged as JR5 in Table 4.

FIELD DUPLICATE. A field duplicate was obtained at INFGW during the March 10 sampling (Table 3) to indicate overall method precision. The project QAPP[2] specifies that the relative percent difference (RPD) of field duplicates should be <30%. All analyte pairs exceeding LOQ (8 of 10 analytes) were within project goals with RPD averaging 8.9%. Duplicate pairs agreed that concentrations were < LOQ for the other two analytes.

LABORATORY REPLICATES. The project goal for laboratory precision in replicate analysis is RPD <20%. The duplicate bottle provided for the April 6 sampling was used to sample the additional V770-TREAT location added between sampling events. A laboratory replicate was run on the EFFGCUA samples as a measure of method precision (Table 3). Average RPD for the four analyte comparisons where both samples exceeded LOQ was 6.0%, and all analyte comparisons were within project goals. In addition, laboratory replicates were analyzed for each of the spiked blanks in both sampling events. The average RPD of 10 analytes in 2 laboratory pairs was 2.0% in batch 1 and 0.4% in batch 2, with all analytes within the project goal of <20% (data not presented).

HOLDING TIME. Samples were stabilized in the field with nitric acid and processing began within several days after receipt at the RTP laboratory on both sampling occasions. Analysis by UPLC-MS was completed within 19 days for the March 10 sampling and within 9 days for the April 6 sampling. This duration is within the holding time of 30 days specified in the QAPP amendment[4] and 1 year specified in the laboratory project QAPP[3].

Table 4. PFAS Concentrations in QC Samples Collected During Sampling at the Solvay Plant in West Deptford, N.J. in ng/L.

			PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFBS	PFHxS	PFOS
Sample Type	**Sample Date**	**Sample ID**	**ng/L**	**ng/L**	**ng/L**	**ng/L**	**ng/L**	**ng/L**	**ng/L**	**ng/L**	**ng/L**	**ng/L**
Field Blanks	March 10, 2021 Batch 1	Trip Blank-1	ND	1.55 U	0.77 U	0.86 U	ND	ND	ND	0.81 U	ND	ND
		Field Blank-1	ND	0.92 U	0.77 U	ND	ND	ND	ND	0.88 U	ND	ND
	April 6, 2021 Batch 2	Trip Blank-2	ND	<LOD	<LOD	<LOD	<LOD	2.43 U	8.85	<LOD	<LOD	ND
		Field Blank-2	ND	<LOD	0.23 U	<LOD	<LOD	<LOD	9.26	<LOD	<LOD	ND
Trip Spiked Blanks	March 10, 2021 Batch 1	Trip spike 50 ng/L-1	45.8	44.4	44.1	43.5	44.4	46.2	49.0	44.7	47.0	52.4
		Trip spike 100 ng/L-1	95.4	84.8	90.3	91.0	91.3	93.7	101	92.0	97.4	108
	April 6, 2021 Batch 2	Trip spike 50 ng/L-2	57.7	59.6	58.1	56.8	56.3	57.4	62.2 JR5	58.4	58.3	58.6
		Trip spike 100 ng/L-2	109	110	109	107	105	104	110	110	108	108
Laboratory Blanks	March 10, 2021 Batch 1	MB	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
		MB_01	ND	1.27 U	0.96 U	0.87 U	ND	ND	ND	1.19 U	ND	ND
	April 6, 2021 Batch 2	MB_01	ND	<LOD	<LOD	<LOD	0.99 U	2.92 U	<LOD	<LOD	<LOD	<LOD

QC Flags:

ND: Non-detect based on no integratable peak area

<LOD: Integratable peak but less than the Limit of Detection

U: Less than the Limit of quantitation, peak area detected but concentration less than the concentration of the lowest standard calibration curve (<5 ng/L)

JR5: Spiked field sample result does not meet QC acceptance criteria for recovery (±20%)